OFFICE OF NAVAL RESEARCH

DEPARTMENT OF DEFENSE FY 1995
DEFENSE UNIVERSITY RESEARCH INSTRUMENTATION PROGRAM (DURIP)

# FINAL PERFORMANCE REPORT

for

07 June 1995 through 06 June 1996

for

Grant No.: N00014-95-1-1127

R&T Project: 1211i09---01

Scientific Officer: Richard G. Brandt
ONR 312

Project Title: Wavelength Tuning in Real-Time, In Situ Analysis of Thin Film Epitaxy

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## EQUIPMENT ACQUISITION AND FABRICATION ITEMIZATION

(Grant No: N00014-95-1-1127, R&T Project: 1211i09---01, P.I.: Wilson Ho)

1. Photoelectron Emission Microscope (PEEM) ........ $13,810.27

   - PEEM Fabrication ........ $10,455.15
     (homemade using the proven design of Prof. Martin Kordesch of Ohio University; machine shop time, electrical feedthroughs, high voltage connections, microchannel plates, power supply, vacuum tilt stage)
   - Precursor Delivery
     Mass Flow Controller ........ $2,389.13
         Tylan General ($1,192.25 + $1,196.88)
     Gas Pressure Sensor ........ $965.99
         MKS Instruments

2. Optical Parametric Oscillator (OPO) ........ $136,189.73

   - OPO Laser Fabrication ........ $79,196.53
     (homemade using components purchased from Newport Corp., CVI, Thor Labs, Virgo, Acton, EG&G, Ranger Scientific, Melles Griot, Edmunds Scientific, Burleigh, Spectral Physics)
   - Ti:sapphire Laser Kit for Pumping the OPO ........ $15,799
         Kapteyn-Murnane Laboratories
   - Diode Pumped Solid State Laser for Pumping the Ti:sapphier Laser ........ $35,000
         Spectra-Physics
   - Storage Oscilloscope ........ $5,353.12
         LeCroy Research
   - He-Ne Laser ........ $841.08
         Uniphase Corp.

**Total DoD/DURIP Grant** ........ $120,000

**Cornell Cost Sharing** ........ $30,000

**Total Cost of Project** ........ $150,000

## III. DESCRIPTION OF INSTRUMENTATION

The two principal items fabricated were the Photoelectron Emission Microscope (PEEM) and the Optical Parametric Oscillator (OPO), which were the two principal items in the original and revised budget. The monies saved in fabricating the PEEM ourselves were used to purchase the Ti:sapphire laser kit and the diode pumped solid state laser for pumping the Ti:sapphire laser. These two lasers are used for pumping the OPO. The layout of the diode pumped solid state laser, the Ti:sapphire laser, and the OPO is shown in Fig. 1. Due to lack of funds from this project, the harmonic generator, shown also in Fig. 1, was not constructed. The harmonic generator, however, is needed for obtaining the wide range of wavelengths required for the research described in this proposal and also in the on-going ONR-DURIP 1996 project *(Title: Femtosecond Spatial Imaging of Charge Transport in Thin Films, Grant No.: N00014-96-1-1041, PR Number: 96PR06658-00, Period: 01 June 96 through 31 May 97, Program Officer: Kenneth J. Sleger, ONR 312, P.I.: Wilson Ho).* The present proposal and the ONR-DURIP 1996 on-going project are intimately tied together; they both require a widely tunable femtosecond laser source and the PEEM. The laser system will provide a broad range of wavelengths (190 nm - 5200 nm) of femtosecond laser pulses, and in fact it is believed to provide the widest range of wavelengths of existing laser systems. Because of this unique capability, it was not necessary to purchase the "Forty-four Wavelength In-Situ/Ex-Situ Ellipsometer" requested in the revised budget. It was believed that the best way to use the available monies was to purchase and fabricate instruments, thus benefiting the two ONR/DURIP projects which form a continuous effort, targeting some common objectives.

In addition to the construction of the OPO laser system, some supporting instrumentation were also purchased. The storage oscilloscope is needed for diagnostics and data acquisition. The He-Ne laser is used for aligning the laser cavity and the PEEM electron optics.

The PEEM was homemade. In the original budget, a commercial unit was requested. However, in subsequent thorough discussions with Prof. Martin Kordesch of Ohio University, it was realized that he had a design which had proven to be effective in carrying out the proposed research and could be built with substantial savings. The revised budget reflected this change. The monies saved were needed for the development of the OPO laser system. The relation of the PEEM (and the ultrahigh vaccum chamber housing it) to the laser system is illustrated in Fig. 1. A closeup of the chamber and the PEEM is shown in the two side views in Fig. 2. A detailed cross section view of the PEEM is revealed in Fig. 3. A small portion of the funds was used to purchase components for delivering precursors required for real-time, in situ analysis of reactions involving precursors with the thin films.

There are a number of important advantages for a homemade PEEM. Our familiarity with the internal parts makes it possible to easily upgrade and modify the microscope in the future. In addition to improving the microscope, the front lens element has been known to become eventually coated with deposits from precursor decomposition and evaporation from sample heating. We will be able to change this lens with minimal difficulties. The OPO system is not commercially available and will allow us to carry out unique experiments.

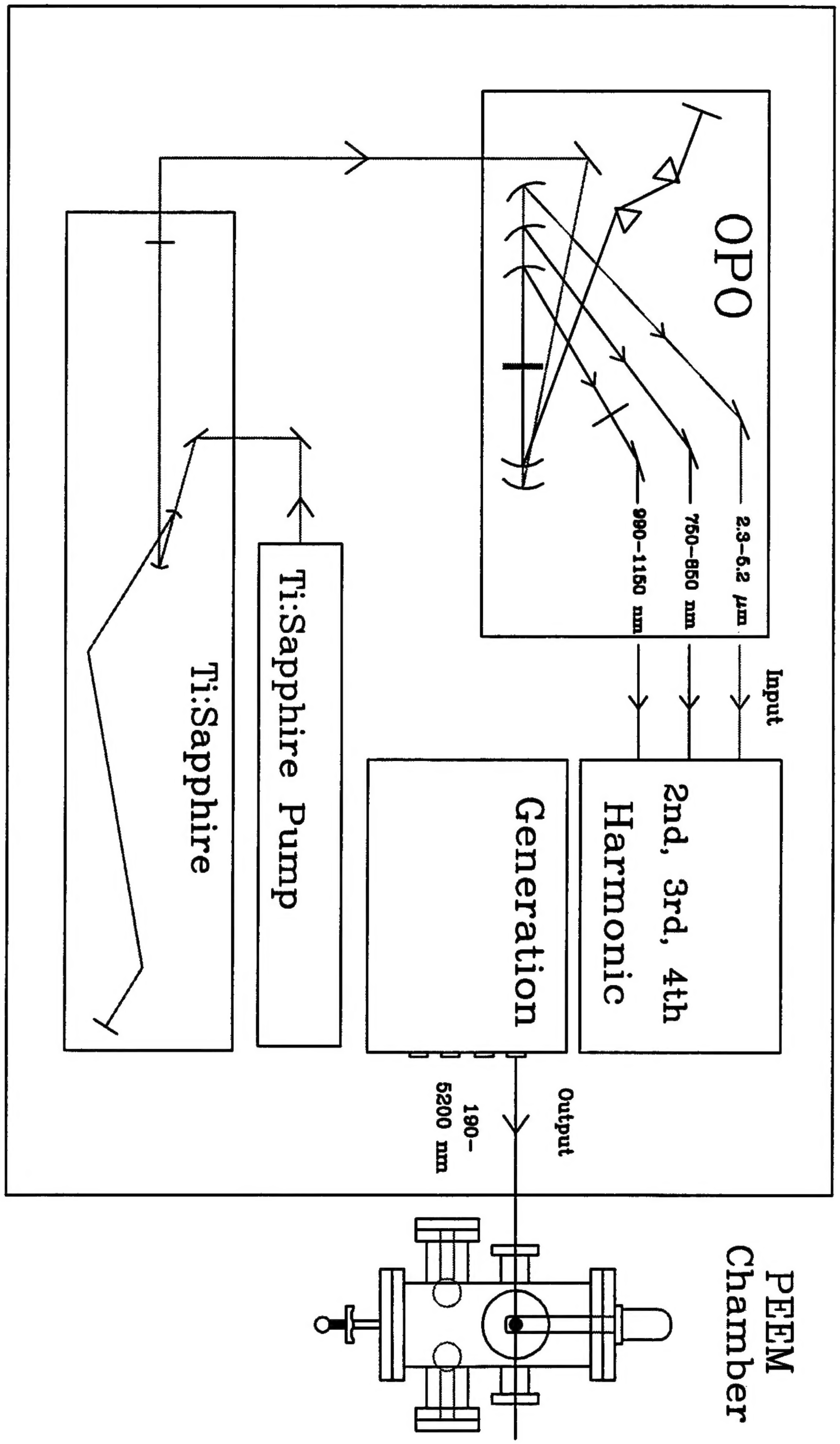
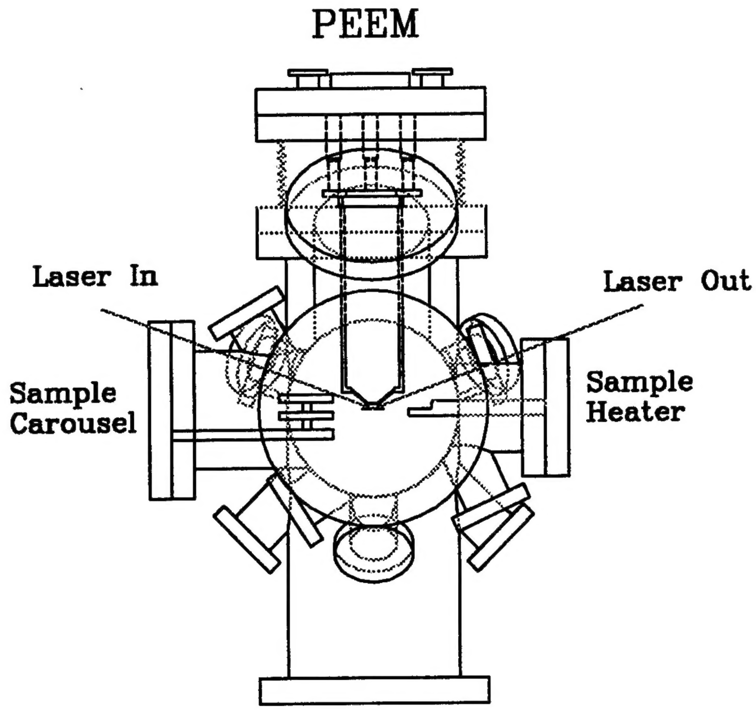



Fig. 1: Top view of laser system and photoelectron emission microscope (PEEM) chamber. The Ti:sapphire pump laser, the Ti:sapphire laser, and the optical parametric oscillator (OPO) are purchased and fabricated with the present grant, as well as the PEEM.



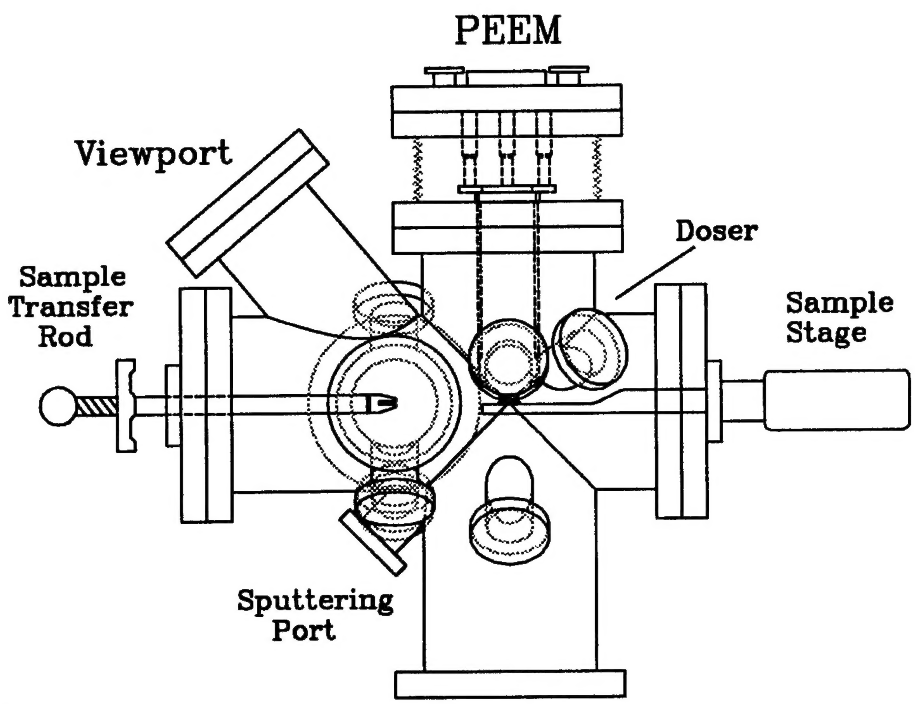




Fig. 2: Side views of PEEM chamber, showing the multiple sample carousel, the sample heater, ion sputtering, sample transfer manipulator, PEEM, sample stage for PEEM measurement, laser input and output, and in situ precursor dosing.

# PEEM

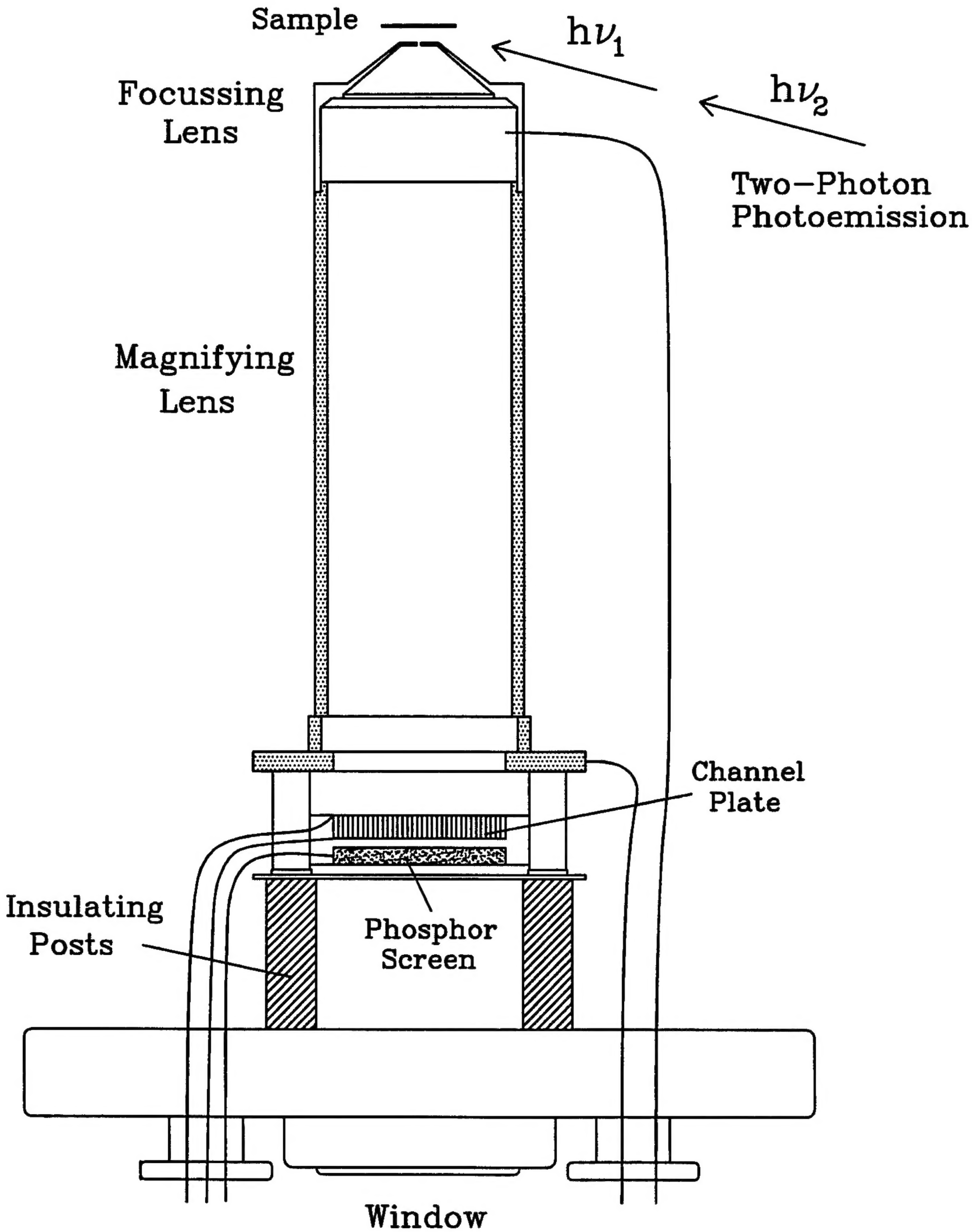



Fig. 3: Detailed cross section view of PEEM. The PEEM was constructed with funds provided by this grant.

## IV. DISCUSSION OF THE USAGE OF INSTRUMENTATION

The proposed research is based on the unique capabilities obtained by combining a broadly tunable all-solid state laser, based on the optical parametric oscillator (OPO), with a photoelectron emission microscope (PEEM). The available wavelengths lie in the deep UV to the mid-IR: 190 nm - 5.2 μm. The PEEM will have an initial spatial magnification of x50 - x100. The femtosecond laser pulses (60 - 90 fs) and the high stability of the OPO provide an unique opportunity for time-resolved spatial imaging of thin films. The instruments provide real-time, in situ analysis of thin film epitaxy, especially the wide band gap semiconductors: GaN, AlN, and SiC.

The primary objective of the proposed research is to understand the properties of thin films and to optimize the growth parameters. The approach is to fabricate and assemble the most effective instrumentation to carry out real-time, in situ spatial imaging of the thin films. The ultrashort laser pulses add another dimension to the analysis, namely time resolution, which allows direct probe of electron dynamics.

The broad tunability of the laser system makes it possible to carry out spatial imaging of the electronic structures of the evolving thin film. Resonant second harmonic generation (SHG) and sum frequency generation (SFG) are all-photon based techniques which allow the investigation of the interaction of precursors with the surface under pressures as high as atmospheres. Operation of PEEM is restricted to pressures lower than about $10^{-6}$ Torr since it is based on photoemission. PEEM, however, provides spatially resolved imaging. The spatial variations in the intensity of the photoemitted electrons are due to spatial variations of the work function, thus providing chemical identification of the evolving thin film. The nonlinear optical techniques (SHG and SFG), although not spatially resolved, yield complementary information on the electronic structure of the surface, including the unoccupied states. This is made possible by the broad tunability of the OPO laser system whose wavelength can be selected to correspond to the electronic transitions.

We are currently setting up the laser system and constructing the PEEM. A new room has been allocated and dedicated to this effort. It is anticipated that the laser system will be assembled and running before the end of 1996. We will also be constructing in parallel the vacuum chamber and supporting pumps. The remaining construction effort is expected to be finished by March 1997. We have already fabricated a metallic pattern on Si(100) at the Cornell Nanofabrication Facility. The smallest feature is one micron. This pattern will be used to determine the spatial resolution of the PEEM. Our research on the growth of GaN, AlN, and SiC is also supported by the ONR/BMDO; our effort uses supersonic jet epitaxy to grow these films on Si. We will first carry out spatial imaging of a grown film and compare the observed morphology with the images obtained by more conventional techniques: optical microscopy, atomic force microscopy, and SEM. These static PEEM experiments will be necessary for calibrating the chemical sensitivity of PEEM and the effects of wavelength tuning on image contrasts. In-situ sputtering will be used to show the real-time, in situ capabilities of PEEM; this sputtering process represents the reverse of film growth.

These initial efforts will be followed by real-time, in situ analysis of supersonic jet epitaxy. Thus our present effort made possible by the DURIP 1995 grant will enhance our on-going ONR/BMDO supported research on supersonic jet epitaxy of wide band gap semiconductors *(Title: Supersonic Jet Epitaxy of Wide Band Gap Semiconductors, Agree ID: N00014-93-1-0499, Mod #: P00007, Period: 01 May 93 through 30 April 99, Program Officer: Colin Wood/Max Yoder, ONR 312, P.I.: Wilson Ho).* Video images of the growth front will be obtained, thus providing detailed information on the growth kinetics and morphology. Therefore, PEEM will complement the other real-time, in situ techniques we are currently employing, namely optical reflectivity, RHEED, and mass spectrometry. One of the objectives of this proposal is to document the strengths and weaknesses of the various real-time, in situ analytical techniques.

In addition to the broad tunability, the OPO laser system provides ultrashort laser pulses (60 - 90 fs). Time resolved experiments are then possible. PEEM based on two-photon photoemission can especially take advantage of the femtosecond laser pulses configured as pump and probe. This opportunity is being implemented in an on-going project: *ONR/DURIP 1996, titled "Femtosecond Spatial Imaging of Charge Transport in Thin Films".* The thin film will be electronically excited by the pump pulse into intermediate electronic states and the time delayed probe pulse then interrogates the transport of the photogenerated carriers in the conduction band. Photoemission takes place with the absorption of the probe pulse since the pump wavelength is chosen to have less energy than the work function. The observed image represents a snapshot of the charge carrier transport in wide band gap semiconductors. Funds from this project will be used to add harmonic generation capability to the existing femtosecond laser system and to provide an upgrade to the spatial resolution of the existing PEEM. Sample processing hardware will also be fabricated. Thus the three projects *(DURIP '95, DURIP '96, and ONR/BMDO grant)* form a continuous and integrated effort in the optimization of the growth and characterization of wide band gap semiconductors by supersonic jet epitaxy. The results from these efforts will impact other research areas of interest to the DOD, including the documentation of the nonlinear optical properties of wide band gap semiconductors, the advancement of a novel technique for transport measurements, and the demonstration and usage of a broadly tunable femtosecond laser system. Time-resolved spatial imaging is a general technique which can be applied to a broad range of materials in addition to the wide band gap semiconductors addressed in our effort.

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**13. ABSTRACT** *(Maximum 200 words)*

This project involves the acquisition and fabrication of instrumentation which combines a broadly tunable all-solid state laser, based on the optical parametric oscillator (OPO), with a photoelectron emission microscope (PEEM). The femtosecond laser system provides output wavelengths in the range 750 - 850 nm, 990 - 1150 nm, 2300 - 5200 nm, and the second, third, and fourth harmonics. Thus a very broad range of wavelengths from the deep UV to the mid-IR is available: 190 nm - 5200 nm. The OPO is homemade and is pumped by a Ti:sapphire laser. The PEEM spectrometer is also homemade and is coupled directly to the laser system for two-photon photoemission experiments. The broad wavelength tunability of the laser system is also being applied to resonant second harmonic generation and sum frequency generation experiments. The ultrashort time duration of the laser pulses (60 - 90 fs) can be used to follow time-dependent processes. The instruments are designed to provide real-time, in situ analysis of thin film epitaxy, especially the wide band gap semiconductors: GaN, AlN, and SiC.

**14. SUBJECT TERMS**
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